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(54) Title: DETERGENT COMPOSITIONS			
(57) Abstract Liquid detergent compositions containing amine oxides, solid builders and optionally bleaches and specified water levels are highly stable. Compositions containing an amine oxide, a builder and a polyalkylene glycol confer easy-iron properties on fabrics.			

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DETERGENT COMPOSITIONS

THIS INVENTION relates to detergent compositions.

Detergent compositions are known which comprise a suspension of a builder in an organic liquid which comprises a surfactant.

5 Non-aqueous compositions of this type of good stability are described in European Patents Nos 30096 and 120,659.

It is however difficult to exclude water completely from detergents, and it is desirable that their water-sensitivity should be reduced.

10 It is believed that a reduction in water sensitivity may aid the dispensibility of such materials ie they may make them easier to introduce into a wash liquid.

Detergent compositions may be introduced into a washing machine by placing them in a unit through which water is passed to carry the detergent with the water into a washing zone.
15 Compositions of low dispensibility may produce solid deposits which remain in the unit. In some cases detergents are placed in containers which are introduced into the washing zone. If the container has restricted access solid deposits may be left in it especially if it was not dry when the detergent was placed in it.
20 It is believed that dispensibility is improved, especially in the latter case, if the water sensitivity of the composition is reduced.

Surprisingly we have found that small quantities of water can be tolerated in such compositions providing that an amine oxide is present. In general the quantity of water should be at most 5 preferably less than 3 and more preferably less than 2 moles per mole for example 0.5 to 1.9 moles of amine oxide and is preferably 1.0 to 1.7 moles of water per mole of amine oxide. By
30 water is meant water which is not included as water of crystallisation of solids, which is irrelevant in this context.

Whilst anhydrous amine oxides are known, in general they are available commercially as aqueous solutions which may contain for example 65% by weight of water. Clearly such solutions are
35 unsuitable for the present purpose and according to one form of this invention means are provided by which they may be converted into a composition useful for inclusion in liquid laundry detergent compositions.

SUBSTITUTE SHEET

This invention comprises a liquid detergent composition which comprises:

- i) a liquid organic phase which comprises an amine oxide;
- ii) a solid builder;
- 5 iii) at most 5 and preferably less than 2 for example 0.5 to less than 2 and preferably 1.0 to 1.7 moles of water (other than water of crystallisation of any solid present) per mole of amine oxide.

The builder may suitably be water soluble and if desired a
10 bleach may be present.

The invention also comprises a composition suitable for inclusion in a liquid detergent which comprises:

- i an amine oxide;
- ii a polyalkylene glycol, which preferably comprises ethylene
15 glycol residues and optionally propylene glycol residues and more preferably polyethylene glycol; and
- iii at most 5 and preferably less than 2 moles, for example 0.5 to less than 2 moles and preferably 1 to 1.7 moles of water per mole of the amine oxide.

20 This composition enables liquid laundry detergents of good stability and easy-iron properties to be produced by the addition of a suitable builder. The weight ratio of polyalkylene glycol to amine oxide is suitably 0.5 to 1 to 2:1.

This composition may be produced by distilling an aqueous
25 solution of an amine oxide in the presence of the polyalkylene glycol, preferably polyethylene glycol, preferably under reduced pressure at a temperature of 40 to 80°C and preferably 65 to 75°C. We have surprisingly found that the presence of the polyalkylene glycol serves to reduce the problem of gel formation which occurs
30 and impedes distillation of an aqueous solution of an amine oxide if it is distilled as such.

We have found that when used together with a builder, polyethylene glycol and preferably another non ionic surfactant amine oxides appear to leave fabrics which have been laundered in
35 them easy to iron.

This invention therefore also comprises a detergent composition for example a laundry detergent composition which comprises:

- i an amine oxide;
- 5 ii a builder;
- iii a polyalkylene glycol, having a molecular weight of 100 to 1500 preferably polyethylene glycol, and preferably a non ionic surfactant other than an amine oxide and preferably a bleach.

10 In many parts of the world it is desirable to reduce the concentrations of phosphates in waste water for environmental reasons. The use of phosphates and particularly condensed phosphates for example sodium tripolyphosphate in detergents has been common for many years but in some cases is now restricted by 15 law for environmental reasons. It is, therefore, desirable to formulate detergent compositions of good performance which are free from phosphates.

This invention also comprises detergent compositions which are free from phosphates and which are suitably laundry detergent 20 compositions which comprise:

- 1) An amine oxide
- 2) A hydroxy carboxylic acid containing 2 to 6 - COOH groups and 1 to 5 - OH groups and
- 3) A polycarboxylic acid comprising at least 10 and preferably 25 at least 100 for example 150 to 2000 and preferably 200 to 1000 - COOH groups and which contain at most 5 and preferably less than 2 moles of water per mole of amine oxide (excluding any present as water of crystallisation).

The polycarboxylic acid may be a polyacrylic acid for example 30 a maleinised polyacrylic acid or is more preferably carboxy methyl cellulose which suitably has 100 to 1000 for example 100 to 500 combined glucose units per molecule and suitably has a degree of substitution (proportion of OH groups which have been converted to carboxy methyl form) of 35 to 85% and preferably 45 to 80%.

35 The hydroxycarboxylic acid may be for example a tartrate or preferably a citrate.

The composition suitably comprises 2 to 10% by weight of component (3) above and preferably comprises 3 to 9% and more preferably 5 to 8% thereof. It suitably comprises 20 to 50% and preferably 25 to 40% by weight of component (2) above.

5 The composition is preferably substantially neutral or alkaline in reaction and it is preferred that acidic substances be neutralised with ammonium, substituted ammonium or preferably alkali metal ions. It is preferred that at least one part by weight of component 3 be present per eight parts by weight of
10 component 2.

It is preferred that liquid compositions should comprise 25 to 70% and preferably 30 to 60% for example 40 to 60% by weight of total solids. The solids preferably comprise a builder which may be present in an amount of 25 to 60% by weight of the total
15 composition. A bleach for example a perborate or percarbonate of an alkali metal for example sodium may be present in an amount of 0 to 25% for example 1 to 20% and preferably 5 to 20% by weight of the total composition. Optionally a polycarboxylic acid comprising at least 10 and preferably at least 100 for 150 to 2000
20 and preferably 200 to 1000 COOH groups which is suitably carboxymethyl cellulose may also be present.

The liquid phase suitably comprises a polyalkylene glycol, preferably polyethylene glycol which is for example 5 to 55% by weight of the total composition and which is preferably 10 to 40%
25 by weight of the total composition and a non ionic surfactant which is suitably 5 to 25% and preferably 7 to 20% by weight of the total composition. The amine oxide is preferably 5 to 100% and more preferably 5 to 50% of the organic matter of the liquid phase by weight.

30 The builder may be for example a condensed phosphate, a silicate or a zeolite or a hydroxycarboxylic acid containing 2 to 6 -COOH groups and 1 to 5 -OH groups which may be citric or tartaric acid. It is preferred however that it be a condensed phosphate which may be an alkali metal, preferably sodium, salt
35 for example sodium tripolyphosphate.

The molecular weight of the polyethylene glycol may be 100 to 1500 and is suitably 150 to 500.

It is preferred that compositions according to the invention be formulated as pourable liquids. To this end it is preferred that the liquid detergent composition should comprise a dispersion of solids in a liquid medium. The solids may be of average particle size at most 10 micro metres. The composition suitably has a pour point of less than 10°C. The pour point may be measured by ASTM test method designation D97-66 approved in 1971. It is desirable that at least 90% of the particles should have particle sizes less than 10 micro metres in diameter. The average particle sizes may be determined by either the number average method or the weight average method. A range of 1.2 to 8 micro metres is preferred.

The surfactant component of the composition may comprise for example a non ionic surfactant. It is suitably an alkylene oxide derivative, the alkylene oxide preferably being ethylene and/or propylene oxide. It may very suitably be a product of reaction of an amine, alkyl phenol or alkanol with ethylene and/or propylene oxide. The alkyl group in the alkyl phenol may be straight chain or branched chain and may contain from 6 to 20 carbon atoms for example six to twelve carbon atoms. The alkanol may contain 6 to 20 carbon atoms and preferably 10 to 16 carbon atom. The alkanol is preferably a primary or secondary alkanol having a linear or mono branched alkyl chain. Suitably the non ionic surfactant comprises a chain of 1 to 10, preferably 3 to 8 alkylene glycol residues, it being understood that this is an average value.

The surfactant may consist essentially of an amine oxide which preferably comprises an alkyl group having 10 to 18 carbon atoms and preferably 12 to 16 carbon atoms and 2 other groups selected individually from the C₁ to C₃ alkyl or hydroxyalkyl group. The composition may if desired comprise two or more non ionic surfactants. The pour point may be adjusted by varying the liquid phase composition, for example, by including surfactants and/or polyethylene glycol of low pour point. Suitably, 10 to 50% and preferably 15 to 30% by weight of surfactant is present. The surfactant may alternatively be anionic or cationic.

Such anionic or cationic surfactants may be of known type for example the anionic detergents may be soaps, alkylbenzene or olefine sulphonates, alcohol sulphates or alcohol alkoxyate sulphates; the cationic surfactants are suitably di-C₁₀₋₂₂ and preferably di-C₁₆₋₁₈ alkyl, di-lower alkyl ammonium salts or hydroxides for example chlorides or sulphates or for example fabric softeners of the C₁₀₋₁₆ alkyl, di lower alkyl (for example methyl), substituted ethyl ammonium salts. The substituent on the ethyl group may be an -OOCR group where R is a C₁₃₋₂₂ alkyl group, and the anion of the salt may be a RSO₄⁻ group where R is lower alkyl for example methyl. The lower alkyl groups are suitably methyl groups.

It is preferred that a polyalkylene glycol, for example a polyethylene/polypropylene glycol or preferably a polyethylene glycol should be present. This contributes to the effectiveness of the compositions in washing fabrics and is of favourable biodegradability. Polyalkylene glycols of average molecular weight 100 to 400 and preferably 150 to 300 are preferred as they tend to reduce the pour point of liquid compositions thus making them more easily usable in a cold environment. Suitably 10 to 50 and preferably 25 to 45% by weight of polyalkylene glycol is present.

Very suitably the composition comprises a bleach which is suitably a perborate or percarbonate of an alkali metal. If desired bleach activators may be incorporated. Suitably 1 - 20% and preferably 2 - 15% by weight of bleach is present.

The composition may also contain additives conventionally found in detergent compositions, for example optical brighteners, ethylene diamine tetra acetic acid, dyes, perfumes and/or enzymes.

In compositions according to the invention the dispersion of the solids in the non-ionic surfactant is remarkably stable especially if the particle size of the solids is at most 10 micro meters. If the solids separate at all then the resulting phase is usually loosely flocculated and is readily redispersible while if the relative amounts of the solids, polyethylene glycol and

surfactant in the composition are correctly chosen there is little phase separation as the liquid fills the space between the loosely packed solid particles.

5 Compositions according to the invention may suitably be made by a process which comprises milling ingredients together.

The percentages by weight given herein are based on the total weight of the composition unless the context requires otherwise.

EXAMPLE 1 : Dehydration of Alkyl Dimethyl Amine Oxide

10 The following procedure produces a 50% w/w solution of an amine oxide having an alkyl group of chain distribution 70%- C12, 27%- C14 and 3%- C16 and two methyl groups substituted on its nitrogen atom (DMAO) in polyethylene glycol (MW200) (PEG 200).

1 equivalent of PEG200 is added to 4 equivalents of DMAO in aqueous solution. (Approx 33% by weight DMAO).

15 The water is removed by distillation at reduced pressure in a 20L Quickfit flask fitted with a N₂ bleed to bottom and a 2 foot froth disengagement column or a splash head, then via a water condenser to a collection vessel and vacuum control.

20 The boiler is charged at no more than 1/3 to 1/2 fill (to give froth space). The contents are agitated at 100 rpm with a stirrer paddle and a nitrogen bleed introduced. A vacuum is applied and the nitrogen bleed adjusted to maintain a minimum flow. When the vacuum is established the temperature is slowly increased to 60-75°C. Frothing can be controlled by fine
25 adjustment of the vacuum or optionally a silicon antifoam at a level of 0.05 to 0.1 equivalents can be added to the reaction mixture.

When the theoretical water has been removed, the water content of the boiler is measured.

30 Distillation conditions,

20L scale, 12 hours, 50 mm Hg at 60°C

500ml scale, 3 hours, 45 mm Hg at 75°C.

Under these conditions, but making a 60% DMAO composition (less PEG 200 being added) the product is a solid at room temperature,
35 the 50% Amine Oxide material being a liquid.

8

60% w/w		50% w/w	
Amine Oxide	61.0	Amine Oxide	50.0
Free Amine	0.7	Free Amine	0.6
H ₂ O	3.5	H ₂ O	5.0
PEG 200	~35.0	PEG 200	~44.5

EXAMPLE 2

Formulation A was produced as follows:

The liquid components were mixed and seven parts by weight mixed with the enzyme and bentonite and the remainder was mixed with the other solid components with a homogeniser and the product milled to a mean particle size of 3 micrometers (weight average). The two mixtures were then mixed together using a homogeniser.

Formulation B was produced as follows:

The components other than the enzyme were mixed using a homogeniser and milled to a weight average particle size of 3 micrometers. The enzyme was then added and mixed in using a homogeniser.

TABLE 1

		Formulation	Formulation
		A	B
5	Sodium Citrate	-	27%
	Sodium Tripolyphosphate	37%	-
	Sodium Carbonate	5%	5%
	Carboxymethyl cellulose ⁺	1%	5%
	Sodium Disilicate	2%	1%
10	"Leukophor" DUK Optical Brightener	0.3%	0.3%
	Enzyme (Protease and amylase)	0.3%	0.3%
	"Synperonic" A3	2.5%	1.3%
	"Synperonic" 87K	7%	3.8%
	Polyethylene glycol, molecular weight 200 (PEG 200)	32%	36%
20	DMAO (as 100%)	10%	7.5%
	Sodium perborate monohydrate	-	10%
	Tetra acetyl ethylene diamine	-	3%
	Bentonite Clay	3%	-
	Water	1%	0.75%

20

⁺ Commercial product sold as "Courlose" A610, degree of substitution 0.5 to 0.7 molecular weight believed to be 60,000 to 100,000.

"Synperonic" A3 and "Synperonic" 87K are the products of condensing respectively an average of 3 moles ethylene oxide and seven moles of a mixture of 92% ethylene oxide and 8% propylene oxide with a mixture of two parts C₁₃ primary alkanol to one part C₁₅ primary alkanol, both having some 2-methyl branching.

The above percentages are all by weight.

The formulations were tested as laundry detergents as follows:

The performance of formulations A and B was compared with that of a commercial detergent as follows. The wash tests were carried out using a Zanussi FL1030 washing machine run at 40°C and 60°C in water with 90 ppm Ca hardness. Standard cloths from EMPA or Krefeld were used to assess the performance of each product. The machine was filled with four test pieces (10x10cm) of each different type of test cloth together with 2 kg of clean cloth as ballast for each wash. Each test cloth was measured in ten different places to assess the increase in reflectance of each cloth. The dose levels of detergent were 60g for formulations A and B and 180g of the commercial product per load.

The results were as follows:

		Formulation				Commercial	
		A		B		40°C	60°C
Cloth		40°C	60°C	40°C	60°C	40°C	60°C
25	EMPA 101	37.8	50.4	40.0	51.0	29.5	45.9
	KREFELD 10C	46.7	52.2	28.8	43.9	33.7	49.7
	EMPA 104	38.6	53.1	33.3	52.4	27.9	46.8
	KREFELD 20C	55.3	59.6	37.4	54.2	31.0	49.1
30	EMPA 111 (Blood)	77.1	78.9	31.5	49.8	77.7	80.9
	EMPA 112 (Cocoa)	25.1	47.8	34.9	47.6	28.3	51.4
	EMPA 114 (Red Wine)	31.7	35.6	49.1	67.2	41.0	43.9
	KREFELD 10K (Coffee)	56.2	59.9	66.9	72.3	56.6	61.4

The numerical values given are the percentage increase in reflectance after washing.

The above results show good performances compared with the

use of three time the weight of a commercial product.

Easy Iron Properties

After using Formulation A for a fortnight, 27 out of 29 people answered yes to the question "Did the clothes handle well during ironing?"

5

A formulation substantially equivalent to formulation A but modified for greater consumer appeal may be made up as follows:

		Weight	%
		kg/te	
10	Synperonic A3	23.828	2.4
	Synperonic 87K	71.567	7.2
	PEG 200	320.452	32.1
	DMAO (as 100%)	97.447	9.7
	Sodium Carbonate	47.958	4.8
15	"Courlose" A610	9.967	1.0
	Sodium Disilicate	18.954	1.9
	Sodium Tripolyphosphate	366.261	36.6
	Ethylene diamine tetra-acetic acid	2.002	0.2
	Optical Brightener	2.998	0.3
20	Blue Colouring	0.029	0.0
	Silicone Antifoam	0.588	0.1
	Bentonite Clay	28.956	2.9
	TiO ₂	2.492	0.2
	Fragrance	3.000	0.3
25	Protease and Amylase Enzymes	3.000	0.3
		<hr/>	<hr/>
		999.500	100.0

EXAMPLE 3

Liquid suspensions comprising the ingredients of the following Table and which comprised about 50% solids were made up. Formulation C was milled to a mean particle size of 3 micrometres by weight average. The other formulations were made by mixing solid pre-ground particles with liquid components the size of the particles not having been determined.

		Formulation A	Formulation B	Formulation C
10	"Dequest" 2016D			
	Phosphonate Builder	-	-	5%
	Sodium Citrate	32%	32%	32%
	Sodium Carbonate	10%	-	10%
	Carboxymethyl Cellulose ⁺	6%	6%	2%
15	Sodium Disilicate	1%	1%	1%
	"Tinopal" CBS-X Optical Brightener	0.2%	0.2%	0.2%
	Enzyme (Protease and amylase)	0.3%	0.3%	0.3%
	"Synperonic" A3	3.75%	3.75%	3.75%
20	"Synperonic" 87K	11.25%	11.25%	11.25%
	Polyethylene glycol, Molecular weight 200	25.5%	25.5%	34.5%
	Tetra acetyl ethylene diamine	-	3%	-
	"Synprolam" 35DMQ	10%	10%	-
25	Sodium Perborate Monohydrate	-	7%	-

⁺ Commercial Product sold as "Courlose" A161, degree of substitution 0.5 to 0.7 molecular weight believed to be 60,000 to 100,000.

"Synperonic" A3 and "Synperonic" 87K are the products of condensing respectively an average of 3 moles ethylene oxide and seven moles of a mixture of 92% ethylene oxide and 8% propylene oxide with a mixture of two parts C₁₃ primary alkanol to one part C₁₅ primary alkanol, both having some 2-methyl branching, and "Synprolam" 35DMO is an amine oxide having an alkyl group corresponding to the said mixture of C₁₃ and C₁₅ alkanols and two methyl groups substituted on its nitrogen atom.

The above percentages are all by weight.

The formulations were tested as laundry detergents as follows:

The performance of formulations A, B and C was compared with that of a commercial detergent as follows. The wash tests were carried out using Miele W451 washing machines run at 40°C and 60°C in hard water (36°C French specification hardness). Standard cloths from EMPA or Krefeld were used to assess the performance of each product. Three machines were filled with four test pieces (10x10cm) of each different type of test cloth together with 2 kg of clean cloth as ballast for each wash. Each test cloth was measured in twelve different places to assess the increase in reflectance of each cloth. The dose levels of the detergent were 60g for formulations A, B and C and 180g of the commercial product per load.

The results were as follows:

		Formulation							
		A		B		C		Commercial	
	Cloth	40°C	60°C	40°C	60°C	40°C	60°C	40°C	60°C
	EMPA101	34.7	30.5	32.8	32.7	24.5	22.1	19.8	18.9
	KREFELD 10C	41.1	53.2	35.9	56.4	29.2	46	42.4	55.3
	EMPA 104	46.5	42.4	46.9	42.8	18.8	19.5	17.1	17.5
	KREFELD 20C	39.8	50.2	52.4	49.7	19.3	20.7	29.5	46.3
	EMPA 111	67.2	67.8	23.9	54.9	78.3	76.3	74.2	73.7
	EMPA 112	40.8	45.6	40.3	35.6	39.8	43.7	38.6	37.2
	EMPA 114	21.7	31.2	31.3	65.0	26.4	29.3	33.2	40.3
	KREFELD 101C	49.3	53.3	52.8	65.8	51.8	53	53.2	57.1
	KREFELD AS12	36.9	36.6	43.7	44.8			49	43.8
	KREFELD PC12	46.4	43.2	50.8	51.4			44.9	42.1

The numerical values given are the percentage increase in reflectance after washing.

The above results show good performances compared with the use of three times the weight of a commercial product.

- 5 The commercial product contained about one third by weight of water and the comparison on a total chemicals content is thus at a ratio of about one to two.

EXAMPLE 4

- 10 Two liquid detergent compositions comprising solids suspended in liquids of the following composition were tested as follows:

	A	B
	w/w	w/w
Sodium Tripolyphosphate	36.7	40.15
15 Bentonite Clay	2.9	-
Sodium Carbonate	4.8	5.00
Sodium Silicate	1.9	2.00
Carboxymethyl Cellulose	1.0	1.00
Sodium Ethylene Diamine Tetraacetate	0.2	0.2
20 Optical Brightener	0.3	0.3
Enzymes	0.3	0.3
Titanium Dioxide	0.25	0.25
Synperonic A3	2.4	2.4
Synperonic 87K	7.2	7.5
25 Polyethylene Glycol Molecular Wgt 200	32.0	40.0
Dimethyl Cocoamine Oxide	9.8	NIL
Perfume	0.25	0.8

- 30 Water was then added to both compositions in amounts of 1%, 1.5%, 2%, 2.5% and 3% by weight.

15

The viscosity after 45 minutes and (where shown) one day at 20°C is shown below:

	Added Water	A		B	
		Viscosity (Poise)		Viscosity (Poise)	
		45 min	day	45 min	day
5	0.0%	48.0		46.0	
	1.0%	50.8		70.8	
	2.0%	60.2		139.0	
	3.5%	90.4	70.9	304.0	340
10	3.0%	800,000.0		706,000.0	

These data show that the amine oxide containing composition was more resistant to degradation by water.

Compositions A and B were also tested for dispensibility in a commercial Zanussi washing machine with a draw dispenser. 2.5 times more residue was left in the tray at the commencement of the washing process in the case of composition B than in the case of composition A.

CLAIMS

1 A liquid detergent composition which comprises a liquid organic phase which comprises an amine oxide and a solid phase which comprises a builder at most 5 for example 0.5 to less than 2 and preferably 1.0 to 1.7 moles of water being present per mole of amine oxide excluding any water present as water of crystallisation of any solid present.

2 A composition suitable for inclusion in a liquid detergent which comprises an amine oxide and a polyalkylene glycol in which at most 5 and preferably less than 2 for example 0.5 to 5 and preferably 1 to 1.7 moles of water per mole of the amine oxide is present.

3 A detergent composition which comprises an amine oxide, a builder and a polyalkylene glycol having a molecular weight of 100 to 1500.

4 A liquid detergent composition which is substantially free from phosphates which comprises an amine oxide, a hydroxycarboxylic acid containing 2 to 6 COOH groups and 1 to 5 OH groups and a polycarboxylic acid comprising at least 10 COOH groups which composition comprises at most 5 and preferably less than 2 moles of water per mole of amine oxide excluding any water present as water of crystallisation of any solid substance present.

5 A composition as claimed in any preceding claim in which the amine oxide comprises an alkyl group having 10 to 18 carbon atoms and two other groups selected individually from C1 to C3 alkyl or hydroxy alkyl groups.

6 A composition as claimed in any preceding claim in which the builder is a condensed phosphate, a zeolite, a silicate or a hydroxycarboxylic acid containing 2 to 6 COOH groups and 1 to 5 OH groups.

7 A composition as claimed in claim 6 in which the builder is a condensed alkali metal phosphate.


8 A composition as claimed in any preceding claim in which the builder is citric or tartaric acid.

- 9 A composition as claimed in any preceding claim which comprises polyethylene glycol of molecular weight 150 to 500.
- 10 A composition as claimed in any preceding claim in which the weight ratio of polyalkylene glycol to amine oxide is suitably 0.5 to 1 to 2 to 1.
- 11 A composition as claimed in any preceding claim which comprises a bleach.

INTERNATIONAL SEARCH REPORT

PCT/GB 92/00943

International Application No.

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC Int.Cl. 5 C11D17/00; C11D1/75		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
Int.Cl. 5	C11D	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹		
Category ¹⁰	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
X	US,A,4 264 466 (K.L. CARLETON ET AL.) 28 April 1981 see column 4, line 35 - column 10, line 31; claims	1,4-8,11
X	US,A,4 874 537 (D. PETERSON ET AL.) 17 October 1989 see column 3, line 22 - column 4, line 4; claims 1-7	1,4,6,7,11
A	EP,A,0 120 659 (IMPERIAL CHEMICAL INDUSTRIES) 3 October 1984 cited in the application see claims 1-8	1,3,4,6-9,11
A	EP,A,0 425 149 (IMPERIAL CHEMICAL INDUSTRIES) 2 May 1991 see page 3, line 3 - line 48; claims; example 6	1,3,4,6-9,11
-/-		
<p>¹⁰ Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"A" document member of the same patent family</p>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
20 AUGUST 1992	27. 08. 92	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	SERBETSOGLOU A. 	

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		Relevant to Claim No.
Category *	Citation of Document, with indication, where appropriate, of the relevant passages	
A	US,A,4 276 205 (M.W. FERRY) 30 June 1981 see column 7, line 1 - column 10, line 10; claims 1-3,19,22	1,3-8,11
A	US,A,3 281 368 (R.E. ZIMMERER ET AL.) 25 October 1966 see column 4, line 12 - line 52; claims	1,3-7,11
P,X	WO,A,9 113 139 (UNILEVER) 5 September 1991 see page 6, line 15 - page 7, line 21; claims	1,5-7,11
P,X	WO,A,9 118 079 (ETHYL CORP.) 28 November 1991 see page 6, line 7 - line 27; claims 1-7; example 1	2,5,10

**ANNEX TO THE INTERNATIONAL SEARCH REPORT
ON INTERNATIONAL PATENT APPLICATION NO. GB 9200943
SA 59604**

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
The members are as contained in the European Patent Office EDP file on
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A-4264466	28-04-81	AT-T- 10011 CA-A- 1148830 EP-A, B 0034387 JP-A- 56159297	15-11-84 28-06-83 26-08-81 08-12-81
US-A-4874537	17-10-89	AU-A- 3673289 EP-A- 0361646 JP-A- 2093000	05-04-90 04-04-90 03-04-90
EP-A-0120659	03-10-84	AU-B- 570035 AU-A- 2604384 CA-A- 1221891 JP-A- 59189198 US-A- 4615820	03-03-88 04-10-84 19-05-87 26-10-84 07-10-86
EP-A-0425149	02-05-91	AU-A- 6461690 JP-A- 3167297	26-04-91 19-07-91
US-A-4276205	30-06-81	None	
US-A-3281368		BE-A- 651634 DE-A- 1518086 FR-A- 1450374 GB-A- 1018774 NL-A- 286242 US-A- 3202714	10-02-65 06-11-69
WO-A-9113139	05-09-91	AU-A- 7344191 EP-A- 0444858	18-09-91 04-09-91
WO-A-9118079	28-11-91	US-A- 5120469 US-A- 5055232 US-A- 5055614	09-06-92 08-10-91 08-10-91

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